Safety practice

Tyre recycling utilising molten metal – risk assessment of a laboratory scale investigation

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Summary

This paper describes the process undertaken prior to the commencement of a proposed tyre laboratory scale experiment carried out in University College Cork, Ireland to identify the hazards, assess the risks, change the design and implement control measures to manage the hazards of the experiment.

Keywords: Laboratory, risk assessment, pyrolysis, tyres

Introduction

In the European Union as in many other countries, employers are legally obliged to carry out risk assessments on occupational health and safety (OH&S)[1]. This legal requirement is, of course, also valid for experiments at tertiary institutions. While it is not unusual for universities to do experimental work, it can be dangerous[2-4]. This may also be true for a proposed tyre laboratory scale experiment, which was carried out in University College Cork, Ireland, as it involved molten metal, flammable gases, flammable solids and nitrogen at temperatures of about 450 °C presenting potentially significant physical hazards such as fire, explosion and splashes of molten metal. **This paper describes how we identified the hazards, assessed the risks, changed the design and implemented control measures to manage OH&S hazards of this laboratory scale experiment.**

Pyrolysis is a promising technology to recycle tyres, as it is capable of recovering the raw materials of tyres. This process uses heat in the absence of oxygen to break the tyres down into pyrolysis oil, gases, carbon black and steel. The amounts and composition of pyrolysis oil, gas and carbon black produced are well known[5-10] although the exact influence of the various operating parameters on the pyrolysis of tyres is still subject to debate as is discussed in three recent reviews[8-10]. Because the chemistry of tyre pyrolysis is well established[5-10], the tyre laboratory experiment is only concerned with the physical separation of the various products (carbon black, steel, vapours) whereas the amounts and composition of the pyrolysis oil and the non-condensable gases are not further investigated. Although tyre pyrolysis is seen as a promising technology, full scale plants have been plagued by being uneconomical. The experiment was designed to verify the concept of a new approach to tyre pyrolysis[11], which would treat whole tyres rather than granulated ones, resulting in potentially substantial energy and capital costs savings.

As will be outlined, the risk of an accident during the laboratory experiment i.e. a fire, injury or worse is negligible, as all reasonable hazards have been identified and preventive measures have been taken. In addition, mitigation measures have been identified, reducing the consequences of an accident. Figure1 gives an image of the experimental setup taken before the heat was applied.

Description of the laboratory scale process

A drawing of the laboratory scale tyre pyrolysis process is presented in Figure 2. The pyrolysis chamber (Figure 2, 2) is manufactured from 6" and 4" ANSI schedule 40 (6" pipe = 7.1 mm wall thickness, 4" pipe = 6.02 mm wall thickness), 316L stainless steel pipes with a 7 mm 316L stainless steel plate welded onto these pipes as the bottom plate. The bottom of the chamber has a slope of 45 degrees. Both legs of the pyrolysis chamber can be opened; leg A by unbolting the schedule 40 flange which ensures that the chamber is airtight, whereas leg B is essentially open to the atmosphere as only

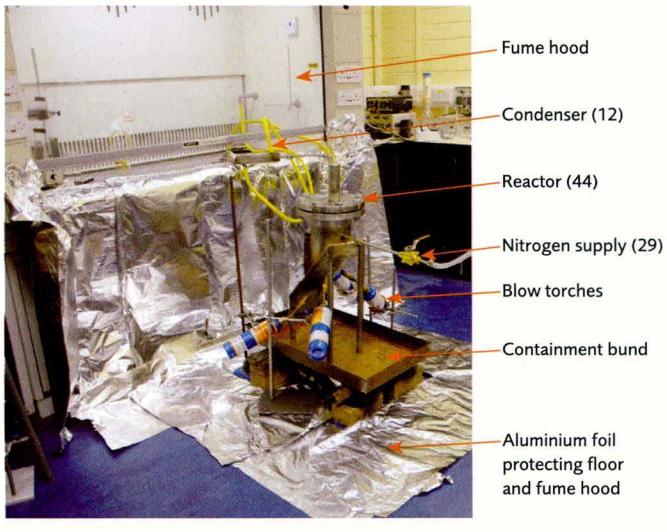

Figure 1: Photo of the experimental setup (refer to the text and Figure 2 for identification of numbers).

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a cover to avoid excess heat loss is provided. The pyrolysis gases are condensed by condenser (Figure 2, 12) with cooling water and collected in a glass beaker (Figure 2, 43). During the pyrolysis experiments, the formation of an explosive atmosphere within the pyrolysis chamber and downstream equipment is prevented by nitrogen inerting the whole system in addition to providing a small nitrogen sweep through the chamber.

The design of the experiment evolved over a number of iterations with the input from various parties i.e. technicians from the department, the manufacturer of the vessels and others, with the result that some hazards were eliminated from the experiment (see Figure 3). For example an earlier design included a charging lock (Figure 2, 27) for tyre pieces allowing adding of materials to the pyrolysis chamber (Figure 2, 2) while at operating temperature. This arrangement, however, made the experiment top heavy as the valves are very heavy designed to operate at 450°C with the risk of toppling the experiment over.

Moreover, unnecessary complexity was removed from the experiment. The original design also included a fan and a cyclone. The cyclone was installed to remove carbon black entrained in the vapour stream originating from the pyrolysis chamber (Figure 2, 3) or leg A. In order to ensure proper functioning of the cyclone, a fan and large amounts of nitrogen would have been required. However removing carbon black from vapour streams with cyclones or bag filters is state-of-the-art and hence proof of this operation is not necessary[11].

Risk assessment

The actual risk assessment procedure used followed a procedure from the University of South Wales[12] and the HSE[13].

According to these two documents, risks must be minimised to the lowest reasonably practicable level by taking measures in the following order.

1. **Elimination**
   Eliminate the hazard: performed as described above.
2. **Substitution**
   Replace the material or process with a less hazardous one: not possible as the materials are essential to the experiment
3. **Engineering controls**
   Install or use additional machinery such as local exhaust ventilation to control the risk: the exhaust from the experiment shall be vented into a laboratory fume hood cabinet.
4. **Administrative controls**
   Reduce the time the experimenter(s) is exposed to the hazard. Perform risk assessments.
5. **Personal Protective Equipment**
   Only after all the previous measures have been tried and found to be ineffective in controlling the risks to a reasonably practicable level, then Personal Protective Equipment (PPE) must be used. However as this will be the first time this experiment is carried out, PPE will be worn regardless of any risk assessments.

In effect, a combination of elimination, engineering controls, administrative controls and PPE was chosen to control the risks of this experiment.

Two different experiments are planned to be carried out on the laboratory scale pyrolysis plant:

Experiment 1: Steel separation with leg A open to the atmosphere; flange (Figure 2, 44) removed, condenser (Figure 2, 12) and pyrolysis oil collecting pot (Figure 2, 43) are not used.

Experiment 2: Rubber pyrolysis with leg A closed, airtight and nitrogen inerted; condenser (Figure 2, 12) and pyrolysis oil collecting pot (Figure 2, 43) are used.

The hazards associated with molten metal were identified from relevant European standards[15, 16], literature[17] and own assessment after a site visit.

In the next section, the potential hazards of the steel separation experiment are assessed, and the additional hazards of the rubber pyrolysis experiment are assessed following that.

Experiment 1: Steel separation risk assessment

During the steel separation experiments, leg A is open to the atmosphere (flange (Figure 2, 44) is removed). Once the operating temperature of 450 °C has been reached, small steel parts will be introduced into leg A to verify that they migrate by gravity from leg A to leg B.

The actual risk assessment of experiment 1 is presented below in tabulated form following the procedure from the University of South Wales[13]:

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No	Hazard	Consequence	Preventive measures	Mitigation measures
1.	*Accidental introduction or accidental spillage of liquids in or onto the molten zinc.*	Ejection of molten metal from evolution of steam or gases. Generation of hot gases and steam.	Only dry steel parts are introduced into the chamber. The exposed surface area of the molten metal is small, minimising the possibility of accidentally introducing liquids into the chamber.	Fire extinguishers suitable for molten metal are provided. Drinking or eating is not allowed in the laboratory. PPE suitable for molten metal / hot gases and steam is worn. Protect area with aluminium foil from possible ejection of molten metal.
2.	*Increased pressure in leg A of vessel.*	Overflow or spillage of molten metal.	Not possible as leg A is open to the atmosphere during experiment 1.	The containment bund can take more than 150% of the entire amount of the molten metal.
3.	*Breakages of bath container i.e. weld by thermal stresses, corrosion or both.*	Leakage of molten zinc through holes.	Very unlikely as the vessel is constructed from a Schedule 40 pipe, 316LSS (minimum wall thickness 6.02 mm). All welds wetted with molten zinc are fully penetrated. Heat up and cool down the containment vessel slowly. The container of the experiment will be exposed to molten zinc for less than 4 hours limiting corrosion. To put corrosion of 316LSS by molten zinc into perspective: A container manufactured from 25 mm thick 316 LSS is suitable for about 8 years of continuous operation in industrial scale hot dip galvanising plants operating at about 450 °C [17]. However at higher operating temperatures the lifespan will be reduced below 8 years[18].	The containment bund can take more than 150% of the entire amount of the molten metal. The molten metal will rapidly solidify on contact with the metal containment bund, as the zinc melt temperature of 419°C is close to the operating temperature of 450°C. The amount of molten metal is as small as possible (less than 2 litre).
4.	*Overheating of zinc.*	Excessive corrosion of the container material (316 LSS) by the molten zinc potentially resulting in leakage. Thermal stresses in container metal of construction resulting in leaks i.e. weld breakage.	The molten zinc temperature is measured by a temperature gauge as the experiment demands that the operating temperature is controlled to 450 °C. Container is over-designed for the duty (schedule 40 pipe). All welds are fully penetrated. As the time frame of the experiment is limited even a temperature overrun of 100 °C would not be a problem.	The experiment is supervised at all times. The temperature will be closely monitored and if necessary the burners will be adjusted to avoid any temperature overrun.
		Inhalation of zinc fumes. Metal fume fever caused by ZnO.	Despite the high operating temperatures of 450 °C, zinc and zinc oxide (ZnO) fumes are either negligible or non-existent: a) The zinc fume concentration in the working environment is low as the zinc vapour pressure is very low at 450 °C (see Appendix). b) The zinc fume concentration in the working environment is further reduced by room and extraction through the fume hood. c) ZnO fumes potentially resulting in 'Metal fume fever' (see explanatory material) are not generated as the operating temperature of 450 °C is too low.	
5.	*Tipping over of the vessel (i.e. unstable, for example, top heavy installation).*	Leakage of molten metal. Burns. Fire.	System is designed such that tipping it over is unlikely. Flammable materials are not stored in laboratory for the duration of the experiment.	PPE suitable for molten zinc is worn. Fire extinguishers are provided. Access to laboratory is limited to authorised personnel only.
6.	*Slip / trip.*	Floor surfaces. Spilled fluids (i.e. water, oil). Inadequate lighting.	Ensure that: a) Trip conditions do not exist. b) Floor is clean / dry. c) Openings are covered or guarded. d) Adequate lighting is provided.	The containment bund can take the entire amount of the molten metal (150% of amount).
7.	*Movement of components or of machinery.*	Collapsing of parts or equipment or materials.	Such activities are prohibited for the duration of the experiment.	A fire watch is provided (two man experiment). Access to laboratory is limited to authorised personnel only.
8.	*Thermal radiation or other phenomena (exposure or ignition by naked flames (i.e. natural gas burners)).* *Heat from burners.*	Fire (ignition of combustible materials).	Protect laboratory furniture closest to the experiment with aluminium foil. Protect Linoleum floor from radiation heat with aluminium foil if necessary. Flammable materials are not stored in the laboratory for the duration of the experiment. Ensure this is the case before proceeding with the experiment.	A fire watch is provided (two man experiment). Fire extinguishers are provided. PPS suitable for molten zinc and thermal radiation is provided.

Shutdown procedure experiment no. 1

a) remove heat source, i.e. switch off natural gas supply;
b) place flange onto leg A;
c) wait until experiment has cooled down to ambient temperature.

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Experiment 2: Rubber pyrolysis risk assessment

With the steel separation experiments concluded, the experiment will be cooled down to ambient and rubber pieces obtained from a waste tyre will be loaded into leg A (see Figure 2). Leg A will be made airtight by bolting down the flange (Figure 2, 44). A pressure test (leak test) will be carried out by covering all connections with soap solution. The entire system will be nitrogen swept for ten minutes to ensure an oxygen free environment before the burners are switched on. For the remainder of the experiment the chamber will be continuously swept with nitrogen. The cooling water to the condenser (Figure 2, 12) will be switched on and a beaker (Figure 2, 43) will be used to collect the condensables. The beaker (Figure 2, 43) will be placed in a laboratory fume hood designed to limit exposure to hazardous or toxic fumes. The non-condensables including the nitrogen sweep will also be vented into this fume hood and extracted to the atmosphere.

In the following table, the additional hazards of the laboratory scale pyrolysis plant due to the presence of the pyrolysis gases and carbon black from the decomposition of the waste tyre rubber are risk assessed.

No	Hazard	Consequence	Preventive measures	Mitigation measures
1.	*Explosive atmosphere inside leg A.* *The European Standard EN 1127-119 distinguishes thirteen types of ignition sources. Of these possible ignition sources, hot surfaces cannot be avoided as they are inherent to the experiment operating at 450 °C. Hence, it is important to maintain an inerted state in leg A during the experiment as otherwise ignition of the hydrocarbons within the chamber may occur.*	Explosion inside leg A. Pressure increase in leg A. Possible overflow of molten metal from leg B. Higher than normal flow of gases through vent line into fume hood.	Nitrogen inertion to prevent explosive atmosphere from forming by: Chamber leg A is initially nitrogen inerted by sweeping it with nitrogen for 10 minutes prior to starting the experiments involving rubber. The flow of nitrogen to the chamber is monitored by the flowmeter. Furthermore, throughout the experiment the chamber leg A is continuously swept with nitrogen, the flow of which is monitored by the flowmeter. The nitrogen flow to the chamber leg A is visually indicated by a flowmeter. Pyrolysis gases are present for a short time (< 1 hour) only reducing the time frame of possible ignition.	The vessel is designed to take the force of an internal vapour explosion without rupture. The maximum explosion pressure of a hydrocarbon – air explosion is 10 bar[20], but the design pressure of the chamber is, according to the manufacturer, in the order of 3,000 psig or 200 barg and is hence capable to take the force of an internal explosion without damage (explosion pressure resistant). The tube and valves are designed to at least 33 bar at the operating temperature[20]; again significantly higher than the maximum possible explosion pressure. The containment bund can take the entire amount of the molten metal, but in the event not all the molten metal will be flowing out.
2.	*Fumes (pyrolysis gases) outside of chamber i.e. due to leak.*	Fire. Smell.	Chamber A will be closed with a schedule 40 flange and a carbon gasket (suitable up to 500 °C) will be fitted, so that fumes cannot escape from leg A.	A fire watch is provided (two man experiment). The experiment will be stopped should there be excessive smell and fumes visible.
3.	*Ignition of hydrocarbon vapours at vent outlet.*	Fire.	The pyrolysis gases are vented into the fume hood and are diluted with air. No ignition sources are present in the fume hood and its exhaust system. The chamber (leg A) and the vent pipe are nitrogen inerted. Moreover pyrolysis gases are present for a short time (< 1 hour) only reducing the time frame of possible ignition.	A fire watch is provided (two man experiment). The collected pyrolysis liquid is unlikely to be highly flammable [6] being similar to diesel. Moreover not more than 100 ml are expected to be collected. In addition the oil is collected in a beaker, which is placed in a laboratory fume hood.
4.	*Hazards of nitrogen.*	Asphyxia.	Only a small flow of nitrogen is used and the nitrogen flow is measured by a flowmeter. Good ventilation is provided in the laboratory. The nitrogen is vented into the fume hood extract system and discharged to a safe location. Leg A and the entire venting system are airtight. The operating pressure is low, minimising leakage rates.	A fire watch is provided (two man experiment).
5.	*Carbon black / pyrolysis gas hybrid mixture or carbon black alone may be flammable at higher temperatures (operating temperature is 450 °C)* [22].	Fire / explosion.	Nitrogen inertion of leg A. Leg A is airtight and leak tested. Leg A will only be opened after experiment has cooled to ambient. Provide flow of nitrogen during cool down period to avoid air from entering leg A of vessel.	The vessel is designed to take the maximum explosion pressure of 10 barg (see above). The maximum explosion pressure of a carbon black / hydrocarbon hybrid mixture is about 8-9 bar[22] and is hence lower than the maximum explosion pressure of a hydrocarbon explosion (see above).
6.	*Increased pressure in leg A of vessel, i.e. due to a closed valve.*	Overflow of molten metal from leg B.	Ensure that all valves are in the correct position before applying heat.	The containment bund can take the entire amount of the molten metal. However, not all the molten metal will be flowing out.

Shutdown procedure experiment no. 2

Normal shutdown: a) remove heat source i.e. switch off natural gas supply;
b) switch off water to heat exchanger;
c) wait until experiment has cooled down to ambient temperature;
d) switch off nitrogen sweep through chamber leg A.

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Figure 2. P&ID of the laboratory scale tyre pyrolysis plant as will be used in the experiment (T = temperature gauge, F = flow meter, = 45 degrees; N2 = nitrogen, H2O = water).

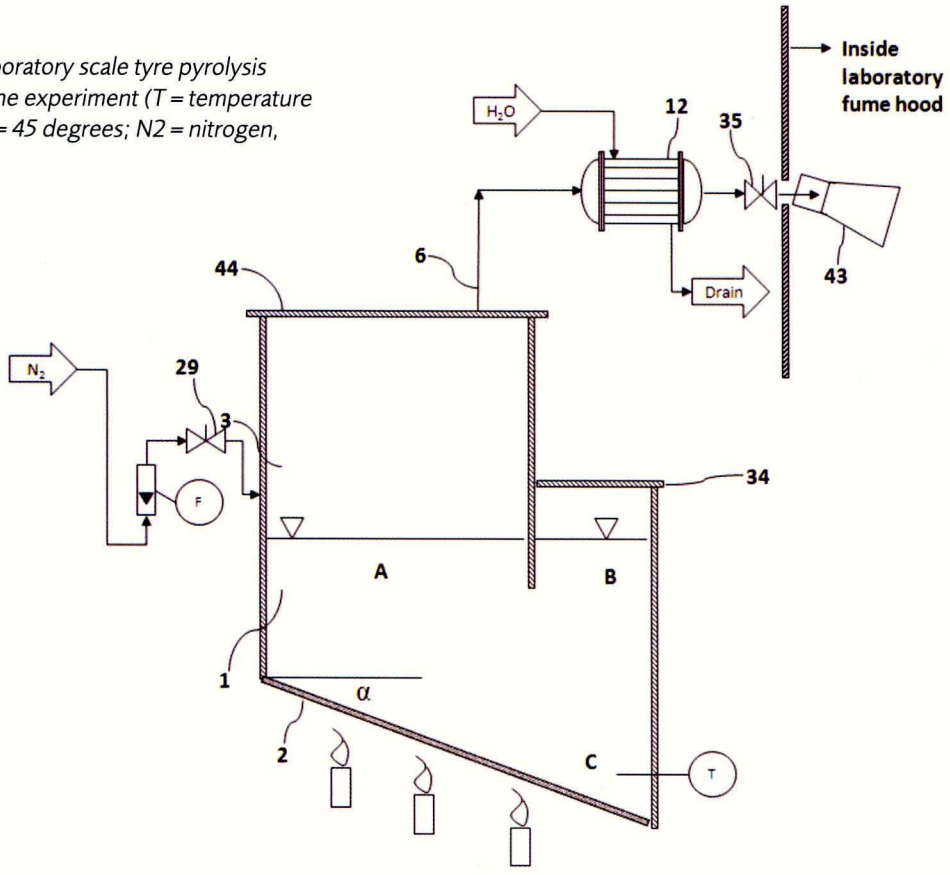
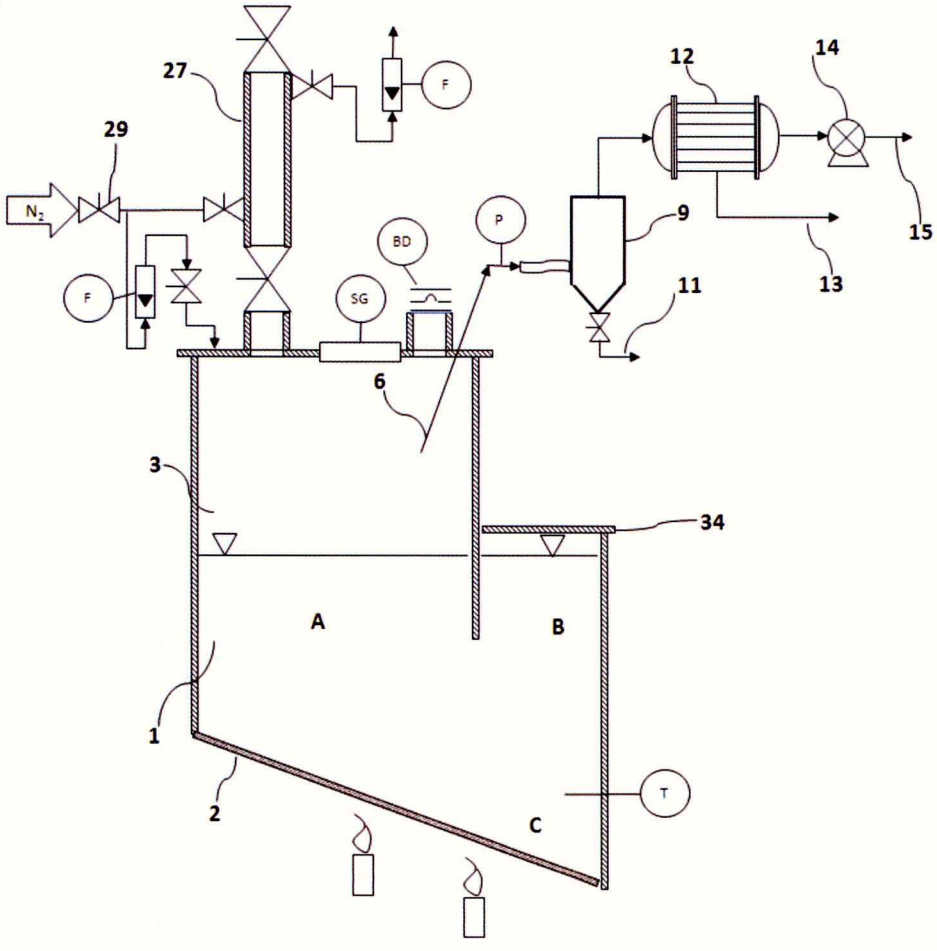


Figure 3. P&ID of the laboratory scale tyre pyrolysis plant as originally planned before applying the risk assessment.



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In Table 3 below the consequences and preventive measures / actions of utility losses are assessed.

No	Utility loss	Consequence	Preventive measures / actions
1.	*Loss of nitrogen.*	Similar to 'explosive atmosphere in leg A' (see above).	Ensure a sufficient amount of nitrogen is available before commencing pyrolysis experiments. Loss of nitrogen will be manually detected by monitoring the flowmeter. Experiment will be stopped and all valves closed should the nitrogen flow fail.
2.	*Loss of natural gas.*	Loss of heat, no safety implications.	The experiment will be stopped / disrupted i.e. chamber will cool down. If the experiment involves rubber, the nitrogen sweep through the chamber will continue until the experiment has cooled down to ambient, preventing air from entering the chamber.
3.	*Loss of cooling water.*	Loss of cooling to condenser would result in an increase of pyrolysis gases being discharged. It may also result in a loss of the water seal due to excessive evaporation and as a result the slight overpressure towards the atmosphere in leg A might be lost.	Loss of cooling water can be detected visually by routing the cooling water return locally into a sink. The amount of rubber in the experiment is limited; hence the amount of pyrolysis gas is also limited. The experiment will be stopped i.e. chamber will be cooled down as described above.
4.	*Loss of electricity.*	The experiment does not require electricity, but the room itself may be dark. Loss of extract from fume hood.	The experiment will be stopped / disrupted. The flow of natural gas to the experiment will be stopped by the operator before leaving the lab. All valves will be closed sealing in the pyrolysis gases. Emergency lights are provided in the laboratory.
5.	*Loss of extraction.*	Pyrolysis gases may enter the room. Smell in laboratory.	Ensure the extract system is in good working order before commencing experiment. The experiment will be stopped as stated above should the fume hood fail. The fume hood is alarmed for failure of extraction.

General safeguards

At the door to the laboratory, the following warnings shall be displayed:

a) high temperature experiment;

b) admittance of non-authorised persons is prohibited.

During the experiments, PPE suitable for molten zinc shall be worn:

a) face protection, gloves, trousers and jacket constructed of self-extinguishing material suitable for molten zinc.
b) safety shoes with a friction/slip resistant sole as used in the chemical industry.

PPE should not be the primary line of defence against accidents[13, 14] and it is not intended to be the case here. The decision to wear suitable PPE was made as residual risks remain and this experiment is executed for the first time.

The following equipment is available at the location to mitigate against an accident:

a) fire fighting equipment;
b) eye wash station / emergency shower;
c) first aid equipment.

Summary of risk assessment outcome

The risk of a major accident, i.e. a fire or injury, is negligible as all reasonable hazards have been identified and preventive measures have been taken. In addition, mitigation measures have been identified and put into place, reducing the consequences of an accident.

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Appendix: Additional explanatory material

Zinc melts at a temperature of 419 ºC and has the following properties in its molten state:

Compound	Density [kg/m3]	Surface tension [N/m]	Viscosity [Pa s]	Melting point [ºC]	Vapour pressure [Pa]
Zinc (molten)	6,508 at 476 ºC	0.755 at 450 ºC	0.003254 at 476 ºC	419	100 at 477 ºC
Water (25 ºC)	1,000	0.072	0.001003	0	3,000

Table 4: Physical properties of molten zinc[23, 24] in comparison to water[24]

Inhaling zinc oxide may result in 'metal fume fever'[25]: Metal fume fever is the symptom of acute ZnO poisoning. Symptoms generally appear within a few hours after acute exposure, usually with dryness of the throat and coughing, and generally resolve within 1–4 days after cessation of exposure without leading to long-term respiratory effects.

Exposure levels leading to the development of metal fume fever have been characterised. While the precursor events for the development of metal fume fever begin to occur at very low zinc concentrations, the condition itself does not fully manifest until exposure levels reach much higher (>75 mg/m^3) levels.

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